CALIBRATION AND TUNING OF THE BALZER'S MASS SPECTROMETER FOR TRACER TESTS PERFORMED IN THE DST USING REFERENCE CALIBRATION BAGS

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1. PURPOSE

This Technical Implementing Procedure (TIP) establishes the methods to ensure that consistent and repeatable analysis of tracer gas concentrations is accomplished by generating qualified reference gas standards, and using those standards to calibrate the Balzer's Omnistar Mass Spectrometer (MS) for the Yucca Mountain Site Characterization Project (YMP) at Ernest Orlando Lawrence Berkeley National Laboratory (LBNL).

2. SCOPE

This procedure applies to all LBNL personnel or contractor personnel following the LBNL procedures who conduct gas concentration tests using the Balzer's Omnistar MS. For all technical activities, data collected using this procedure, and any equipment calibrations or recalibrations that may be required, shall be in accordance with this technical procedure and in full compliance with YAP-12.3Q, *Control of Measuring and Test Equipment and Calibration Standards.*

This procedure is designed to provide detailed methodology to assure consistent calibrations of the Omnistar MS. If this procedure cannot be implemented as written, YMP-LBNL personnel shall notify the responsible Principal Investigator (PI) or designee. If it is determined that a portion of the work cannot be accomplished as described in this TIP, or would produce undesirable results, that portion of the work shall be stopped and not resumed until this procure is modified per YMP-LBNL-QIP-5.2, *Preparing Master Planning Documents & Quality/Technical Implementing Procedures*.

If the responsible PI or designee determines that a modification or a revision to the TIP would cause an unreasonable delay in proceeding with the task, then an expedited change to the procedure, including documentation of deviation from the approved procedure, can be made according to YMP-LBNL-QIP-5.2. Such changes are subject to review, usually after the task has proceeded, and thus work performed under TIPs with expedited changes is done at risk of future invalidation.

Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used. When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

3. PROCEDURE

The following procedures shall be followed to prepare gas reference standards and to calibrate the MS. Section 3.2 discusses the preparation of reference gas storage bags. Section 3.3 is followed to fill a reference bag directly from a cylinder while Section 3.4 is used to prepare dilutions from a cylinder.

3.1 Specialized Equipment Requirements

3.1.1 Reference Gas Cylinders

Q cylinders are obtained through vendors that are on the Qualified Supplier List. If Q cylinders are not available, a calibration may still be performed which can provide concentration relative to the initial cylinder gas concentration. This calibration will not give absolute concentration, but only a relative concentration, which is acceptable for many types of gas tracer tests. Data from calibrations using non-Q gas cylinder shall be reported in units of C/C_0. Calibrations using Q gas cylinders may be reported in absolute units. The cylinder type and its Q-status shall be referenced in the scientific notebook.

3.1.2 Gas Storage Bags

5 liter gas storage bags are used for holding reference gas samples. A recommended manufacturer is Calibrated Instruments Inc. Fill the 5 liter bags with no more than approximately 1.2 liters of gas to prevent damage to the bag in transit.

3.1.3. Stec sgd-710c Gas Divider

A Stec sgd-710c gas divider is used for producing dilutions of tracer gas. Since this unit is not directly traceable to the National Institute of Standards and Technology, the gas divider must be calibrated using calibrated Mass Flow Controllers (MFCs). See Attachment 1 for calibration of the Stec sgd-710c.

3.1.4 Calibrated Mass Flow Controllers (MFCs)

MFCs in the ranges from 0-100 standard cubic centimeters per minute (SCCM) to 0-10 standard liters per minute (SLPM) shall be used to verify the calibration of the Stec sgd-710c gas divider. They can also be used to produce dilutions of gas. The serial number and calibration due date shall be entered in the scientific notebook for each MFC utilized.

3.2 Preparing Bags to be Filled

A. Verify by visual inspection that the integrity of the bag is not compromised. Reject any damaged bags.

B. Evacuate the bag using a Mityvac Model ld109 hand pump or equivalent, which is attached to the reference bag using a Leur fitting. Pump the bag until the vacuum gage reads greater than 20.0 cm Hg. (Note: this is a non-Q measurement, which shall use the visual gage that is connected to the Mityvac hand pump. The gage should be checked so that there is no visible zero shift and periodically its performance should be verified by pulling a hard vacuum to ensure that its indicator will go through its full scale range.) The bag should be able to maintain this vacuum for 1 minute. Bags that cannot maintain a vacuum for 1 minute shall be rejected.

C. Mark each bag with a permanent marker to identify tracer type and concentration as well as date of filling.

D. Record the bag number, tracer type, concentration, source cylinder(s), dilution, fill date and time, fill volume, and personnel present into a scientific notebook.

3.3 Filling from a Cylinder with no Dilution.

A. Identify the cylinder to be used and install the dedicated pressure regulator for that compound.

B. Connect a length of 1/4" poly-flo or other similar plastic tubing to the regulator with a compression fitting.

C. Close the regulator's outlet valve and open the cylinder's valve, leak check all connections with a snoop or a soap solution.

D. Connect a 0-1 SLPM MFC to the poly-flo tubing and set the MFC to 300 SCCM.

E. Adjust the regulator on the gas cylinder to deliver gas flow to the MFC.

F. Let the system purge itself for 5 minutes.

G. Connect a prepared bag to the outlet of the MFC using a poly-flo to Leur adapter.

H. Connect a prepared bag to the Leur adapter outlet for 4 minutes, for a total fill volume of 1.2 Standard Liters.

I. Record all fill data in a scientific notebook.

J. Close cylinder valve.

3.4 Making Gas Dilutions with the Stec sgd-710 Gas Divider

A. Check the Calibration on the Stec sgd-710 using calibrated MFCs as outlined in Attachment 1. Record the flow rates for the Balance and Component gas streams in a scientific notebook for all dilutions that will be used at the Component and Balance pressures that will be used during the generation of the gas reference bags.

B. Identify the cylinder to be diluted and install the dedicated pressure regulator for that compound.

C. Identify the zero air cylinder and install the dedicated pressure regulator.

D. Connect the cylinder to be diluted to a 1/4" compression fitting at the rear of the gas divider marked COMP (component gas) using poly-flo tubing.

E. Connect the zero air cylinder to the 1/4" compression fitting at the rear of the gas divider marked Balance. Use 1/4" poly-flo tubing.

F. Connect a short length of 1/4" poly-flo tubing to the gas divider fitting marked OUTLET. Install a barb to Leur adapter on the other end.

G. Turn selection valve to the "0" position and open the component cylinder valve and set the regulator to about 18 PSIG. Note: no gas should flow from the cylinder at this time. If gas does flow then dirt contamination of the gas divider should be suspected and the instrument shall be treated as an out of calibration instrument as directed by YAP-12.3Q.

H. Open the zero air cylinder valve and set the regulator to about 10 PSI. Note: exact pressures are not critical but the zero air pressure must be kept below the component gas pressure.

I. Leak check all connections with a snoop or a soap solution at this time.

J. Set the selection valve to the desired dilution, wait twenty seconds for the flow to stabilize and read the flow rate from the visual indicator at the front of the gas divider. (Note: this is a non-Q measurement.)

K. Based on the flow rate observed, fill the bag for the amount of time required to fill the bag to a volume of 1.2 liters.

L. Record all fill data in a scientific notebook.

M. Repeat steps 3.4.J to 3.4.L for each different dilution required.

N. At the end of use, set selection valve to "90" and turn off the component gas cylinder valve. Allow the pressure in the component gas regulator to reach 0 PSI. Turn zero air cylinder valve off and allow its regulator to reach 0 psig. Disconnect cylinders from the gas divider and store the divider in a clean place.

O. Make sure cylinders are closed and not leaking.

3.5 Tuning the Mass Spectrometer

Two aspects of the Omnistar MS must be tuned, the ion source, and the mass scale. Pull down menus, associated with the software that is used to operate the MS are often referred to. An underscore will be used to indicate reference to a pull down menu.

The MS must be turned on and pumped down, and the SEM & Emission must be turned on for at least 1 hour before proceeding to the next step.

3.5.1 Tune Ion Source (filament)

Use the software Tuneup.exe that is provided with the QS421 software package. From the Tune Menu choose IonSource. The

Tune Ion Source QMA200 window opens. Presently there are three ranges which are enabled, #0 (mass range 80-90), #1 (mass range 120-135), and #2 (mass range 24-32). To view the output from a channel click on Display, then select MeasureData. Ion source parameters may be changed and the effect on the output may be viewed in real time. Follow the discussion of "Optimizing the Ion Source Parameters Omnistar" (Attachment 2). The software used is part of the measuring and test equipment and will be controlled by the process described in YAP-12.3Q.

3.5.2 Tune Mass Scale

The mass scale is tuned in several steps. First the overall scale must be adjusted (Tune Mass Scale), then each compound must be fine tuned (Calibrate Mass Scale)

A. Tune Mass Scale

With the MS pumped down and warmed up, start the Tuneup.exe program. Choose Tune then choose QMS 200 Tune Mass Scale. The mass scale is tuned with a high concentration (10PPM or greater) of the compound of interest. Connect the gas to be tuned to the inlet of the MS and using the mouse pointing to the graph click to obtain a vertical line. Using the arrows move the peak until it is as close as possible to the correct peak location. The peak can be made sharper by adjusting the Resolution parameter.

B. When the mass scale has been adjusted then each of the compounds of interest must be fine tuned. Leave the compound of interest connected to the inlet of the MS, close the tuneup.exe window and execute measure.exe. Choose calibration. Then for mass scale choose the compound connected (e.g. SF_6.msp). Choose Coarse, then OK. Now the MS will adjust the peaks. Repeat this again using the Fine adjustment. If the program returns the indicator "calibration failed," rerun the mass scale calibration, several times if necessary. If still unsuccessful choose parameter, and under mass reduce the Resolution.

C. For each compound of interest, repeat the Mass Scale Calibration but do not retune the mass scale.

3.6 Calibrating the Mass Spectrometer using Standard Bags-

A. Use the measure.exe program to record the data to the laptop computer. Make sure that Save Cycle Data has been chosen before starting the calibration. Record in a scientific notebook the name of the personnel, data file name, and the time data collection has started.

B. Using the Calibrated Standards Bags created in Sections 3.3 and 3.4, sequentially attach the bags to the MS. Start with a zero air reference bag and allow the ion current to reach a steady state for the compound of interest. If the MS has been exposed to high concentrations of the calibration compound, it may take up to an hour to reach steady state.

C. Sequentially replace the bags, going from lower to higher concentration. It will take between 5 to 15 minutes to reach a steady state ion current for each concentration. Record the time, cycle number, and concentration in the scientific notebook when each gas standard bag is attached to the MS.

3.7 Scientific Notebook Requirements

A scientific notebook record log for calibrating the mass spectrometer shall include at a minimum:

A. Calibrating personnel.

B. MFC and Stec-sgd-710c serial numbers and calibration due date.

C. Flow rates and pressures used for filling gas sample bags.

D. Tracer bag concentrations and identifiers for all reference bags used or filled.

E. Time, cycle number, and sample bag identifier for each sample bag attached to the MS.

F. When checking calibration of the Stec-sgd-710c according to Attachment 1, include selection valve position and flow rates of the mass flow controllers.

G. Time at which and method used to ensure the completeness and accuracy of the data has been established and the method by which the security of the data is maintained

4.0 Records

4.1 Lifetime

The data generated from calibrating and tuning the MS is stored on the hard disk of the computer attached to the MS. The data shall be backed up at least daily. The data generated by following this procedure and the scientific notebook used in MS calibration is used for converting tracer test data from ion current format, to qualified absolute concentration or relative concentration units. The calibration data collected under this TIP shall be submitted with a TDIF and gas analysis test data, which are processed using the calibrations generated by this TIP. The related data shall be turned over to the Technical Data Coordinator in accordance with YMP-LBNL-QIP-SIII.3, *Submitting Key Data to the Yucca Mountain Project Office,* and AP-SIII.3Q, *Submittal and Incorporation of Data to the Technical Data Management System* for submittal to the Technical Data Management System (TDMS).

4.2 Non-Permanent

None

4.3 Controlled Documents

Technical Implementing Procedure

4.4 Records Center Documents

Records associated with this procedure shall be submitted to Records Processing Center (RPC) in accordance with AP-17.1Q, *Record Source Responsibility for Inclusionary Records*.

5. RESPONSIBILITIES

5.1 The Principal Investigator (PI) or designee is responsible for assuring full compliance with this procedure. The PI shall require that all personnel assigned to work to this procedure shall have the necessary qualifications and training to adequately perform the procedure; and they shall have a working knowledge of the LBNL QA Program. When procedure-specific responsibilities are to be delegated to contributing investigators or other personnel, the details of these responsibilities are as stated in this procedure.

5.2 Staff Members are responsible for following this procedure and turning over related documentation to the Records Coordinator for submittal to the

Records Processing Center in accordance with AP-17.1Q.

Special qualifications and/or training unique to the conduct of this procedure are as follows: In the acquisition phase of the project, field supervisors and/or managers (or their designates) shall have a working knowledge of mechanical and electronic equipment. Field personnel shall have all safety training as required by LBNL Environmental Health and Safety regulations to operate basic electrical and low pressure compressed gas systems, as well as to be in compliance with ESF General Underground Safety Training requirements.

6. ACRONYMS AND DEFINITIONS

6.1 Acronyms

DST	Drift Scale Test
ESF	exploratory studies facility
Kpa	kilopascal
LBNL	Lawrence Berkeley National Laboratory
MFC	mass flow controller
MS	Mass Spectrometer
PC	personal computer
PI	Principal Investigator
PSIG	pounds per square inch, gage
QA	Quality Assurance
QARD	Quality Assurance Requirements and Description
SCCM	standard cubic centimeters per minute
SLPM	standard liter per minute
TDMS	Technical Data Management System
TIP	Technical Implementing Procedure
YMP	Yucca Mountain Site Characterization Project

6.2 Definitions

Calibration: The process of establishing the accuracy of a standard or measuring device, which may require resetting parameters on the device to improve its accuracy.

Staff Member: Any scientist, engineer, research or technical associate, technician, or student research assistant performing quality-affecting work for YMP-LBNL.

Technical Implementing Procedure: Each TIP describes YMP-LBNL technical tasks that (1) are repetitive, (2) are standardized, and (3) can return different results if deviation from the sequence of steps occur.

7. REFERENCE

AP-17.1Q, *Record Source Responsibility for Inclusionary Records*

AP-SIII.3Q, *Submittal and Incorporation of Data to the Technical Data Management System,*

Operating manual: OmniStar / ThermoStar GSD 300 O, GSD 300 T; *Gas Analysis System*

YAP-12.3Q, *Control of Measuring and Testing Equipment and Calibration Standards*

YMP-LBNL-QIP-5.2, *Preparing Master Palnning Documents & Quality Technical Implementing Procedures*

YMP-LBNL-QIP-SIII.3 *Submitting Key Data to the Yucca Mountain Project Office*

8. ATTACHMENTS

Attachment 1 Checking the calibration of the Stec sgd-710c

Attachment 2 Optimizing the Ion Source Parameters OmniStar

9. REVISION HISTORY

09/30/98 Revision 0, Modification 0:

This is the initial issue of this procedure.

08/31/99 Revision 0, Modification 1:

Modification to eliminate reference to the cancelled YMP-LBNL-QIP-12.0, reference YAP-12.3Q and include Attachment 2 to incorporate Initial start up tuning requirements referenced in the OmniStar Operating Manual. Minor editorial corrections.

10. Approvals

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